

RESEARCH ARTICLE

Use of the modified controlled atmosphere cone calorimeter for the assessment of fire effluents generated by burning wood under different ventilation conditions

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Summary

The ISO 5660-1 cone calorimeter is an affordable, practical, and commonly used solution for the measurement of main fire properties of products and materials. Among its chief drawbacks is its limited ability to reproduce combustion conditions found in real fires. This deficiency is mainly due to its inability to control oxygen availability in order to simulate an underventilated fire. As several toxic or potentially toxic species are formed primarily in oxygen-poor conditions, the controlled atmosphere cone calorimeter (CACC), now defined in ISO 5660-5, is a major improvement when trying to study the toxicity of fire effluents. A proposed additional modification of the CACC via the introduction of chimney sampling ports and oxygen sensors improves the reproducibility and veracity of effluent sampling. This approach allows the implementation of various techniques to sample, collect, and analyze the generated fire effluents. In this study, the experimental set-up was used to capture fire effluents generated by burning wood under different ventilation conditions. A gas chromatograph coupled with mass spectrometer was used to assess and compare the chemical composition of the collected samples. The results obtained with the new experimental set-up proved the ability of the system to reproducibly generate fire effluents under various controlled burning circumstances. It could prove useful as a tool in characterizing the toxicity of fire effluents from various materials on a benchtop scale and ultimately contribute data for the numerical modeling of toxicity of fire effluents in real buildings.

KEYWORDS

cone calorimeter, fire effluents, wood combustion

1 | INTRODUCTION

In fire safety engineering, the emphasis is primarily put on structural safety of buildings during fire, the possibility of evacuation of people in the building after the fire has started, as well as safety of the firefighters in the intervention, and the effect of a burning building to its

immediate neighborhood in terms of transfer of fire from the burning building to the neighboring building. Although the fact that most casualties in fires are related to toxic fire effluents is well supported in the literature, not much has been done on the legislative approach to the problem. One of the reasons for this situation is the lack of a simple, fast, and affordable, yet reliable method for evaluation of the toxicity

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of fire effluents. Therefore, it is very important to understand the generation of fire effluents and their composition in assessing fire-related risks in buildings or other engineering structures, such as tunnels.

The use of fire-related performance-based design in building construction has been increasingly supported by the progressing reliability and capability of numerical tools for simulation of various fire scenarios. The results, however, strongly depend on the quality of the input data for models. Not many tools are able to quickly and accurately model the dynamics of creation and mobility of individual compounds in these effluents. This is often due to the lack of data on the effect of rapidly changing conditions on the underlying processes. At the moment, little weight is given to possible effects of toxic smoke transfer from the burning building to its immediate surroundings.

There is data available that the production of toxic gases in the first phase of a building fire originates mainly from the furniture and not from the building itself.¹ Although this is a reasonable expectation in cases where the combustible parts of the building's structure are protected with noncombustible layers, this assumption may not be true where the material is exposed or covered with a thin combustible layer. In particular, linings should have their smoke release addressed with respect to toxicity of the smoke as they might also contribute toxic gases in the early stage of fire. Currently, this is not the case within the EU legislation, mainly due to no reproducible way of determining smoke toxicity.

Bench scale tests present a fast, affordable, and practical solution in fire testing, provided that the results can be linked with real fire scenarios. There are several bench top tests available that are used to assess the toxicity of combustion products. Among these tests are smoke density chamber according to EN ISO 5659-2, steady-state tube furnace test ISO TS 19700, and cone calorimeter EN ISO 5660-1. None of these tests can truly replicate the combustion conditions that are found in real fire test or in the room corner test ISO 9705,² which is accepted as the smallest "real scale" test.³ This deficiency is attributed to the fact that in real fires, the oxygen availability is the controlling factor: an underventilated fire is difficult to replicate because of the variable balance between the supplied and consumed oxygen.⁴ While the steady-state tube furnace provides the means of controlling oxygen availability in its standard, the rest of common apparatus lacked such provisions. In order to better explore underventilated conditions, various modifications of the ISO 5660-1 apparatus were scrutinized, settling on a modification that is now standardized in ISO 5660-5.⁵

Most of the research focuses on the description of standard cone calorimeter parameters, in particular, heat release rate and smoke production rate. Only a few studies use the cone calorimeter for the toxicity and ecotoxicity assessment of fire effluents of various types of fuel. In some cases, the use of an open cone calorimeter, according to ISO 5660-1, is reported.⁶⁻⁸ In other cases, the researchers have

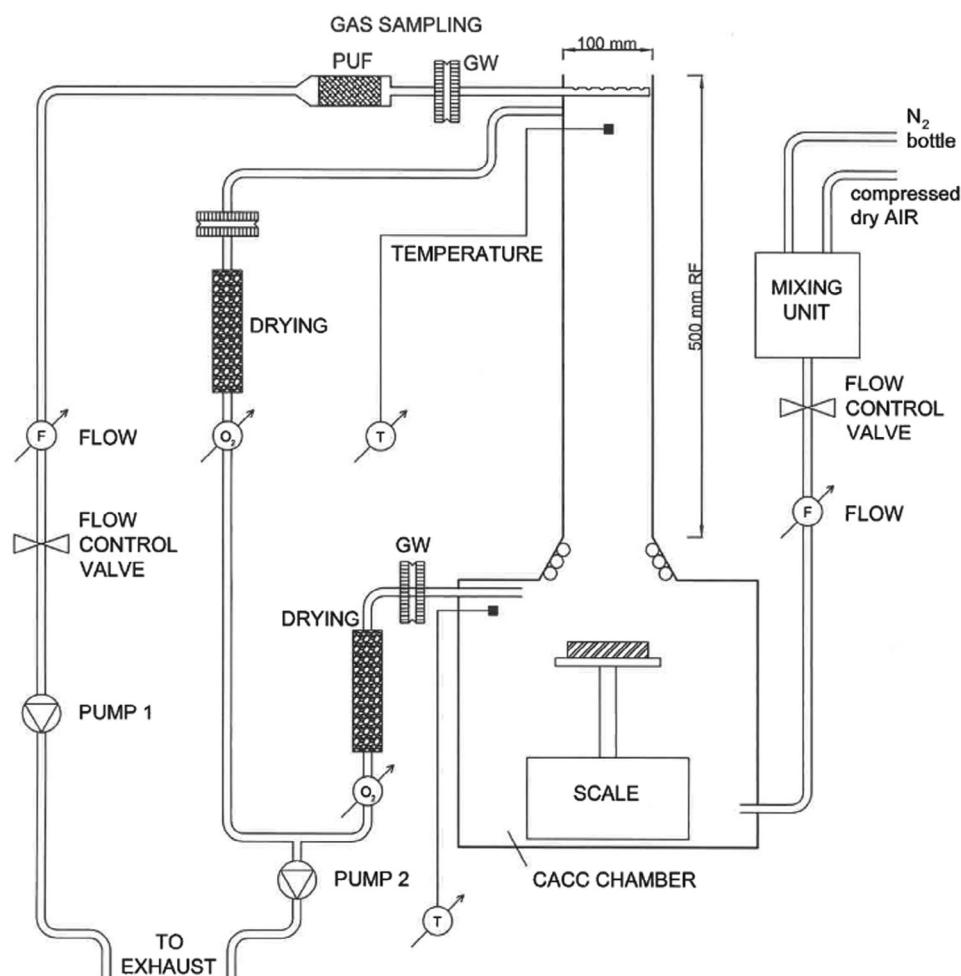


FIGURE 1 Schematic presentation of the experimental setup

been using various modifications of the cone calorimeter to control the atmosphere in the chamber where the burning takes place.⁹⁻¹³

In our experiment, the standard controlled atmosphere cone calorimeter forms the base, which was modified in order to get additional experimental information. The controlled atmosphere calorimeter is essentially a chamber into which a gas mixture of air and nitrogen is led with the standard conical heater at the top of the chamber. Other gases, such as carbon dioxide or water vapor, remain uncontrolled aside from feedgas selection. The whole sample stand and the scale for measuring the test specimen mass are put in the chamber during the experiment. Early alternative to this solution was completely enclosing the cone calorimeter in a controlled atmosphere box, but this was found too impractical.¹⁰ The attached chimney limits the availability of oxygen, leading to conditions in the real fire case being better imitated in the central part of the combustion gases as well as avoiding or limiting after-burning with ambient air.^{14,15} Although the

apparatus comes significantly closer to representing large fire conditions compared to alternative benchtop solutions, it is noted that the conditions cannot be replicated perfectly. The intensity of chemical reactions is affected with both the availability of the reagents (in this case oxygen) and the thermal conditions, mainly the temperature of the gases. The latter was not controlled in our experiment or the standard, although the addition of some thermal insulation or an active temperature control system could provide for a degree of control of desired thermal conditions as well. The main addition to the standardized apparatus were additional sampling ports in the stack in order to better measure oxygen condition and allow for efficient and representative effluent collection.

Several sampling systems are in use to collect and store effluents.^{16,17} Polyurethane foam (PUF) plugs are a good performing and practical choice when focusing on volatile and semi-volatile organic compounds with continuous sampling.^{18,19}

TABLE 1 Sample names and designations

Oxygen level	21%	15%	10%
Spruce	PI1, PI2, PI3	PI4, PI5, PI6	PI7, PI8, PI9
Particle board	PB1, PB2, PB3	PB4, PB5, PB6	PB7, PB8, PB9
OSB	O1, O2, O3	O4, O5, O6	O7, O8, O9

2 | AIM

The aim of the experiments was to explore the capability of the modified cone calorimeter to allow the reproducible creation of fire effluents under controlled burning circumstances. The ability of the new sampling system to capture the generated fire effluents, combined

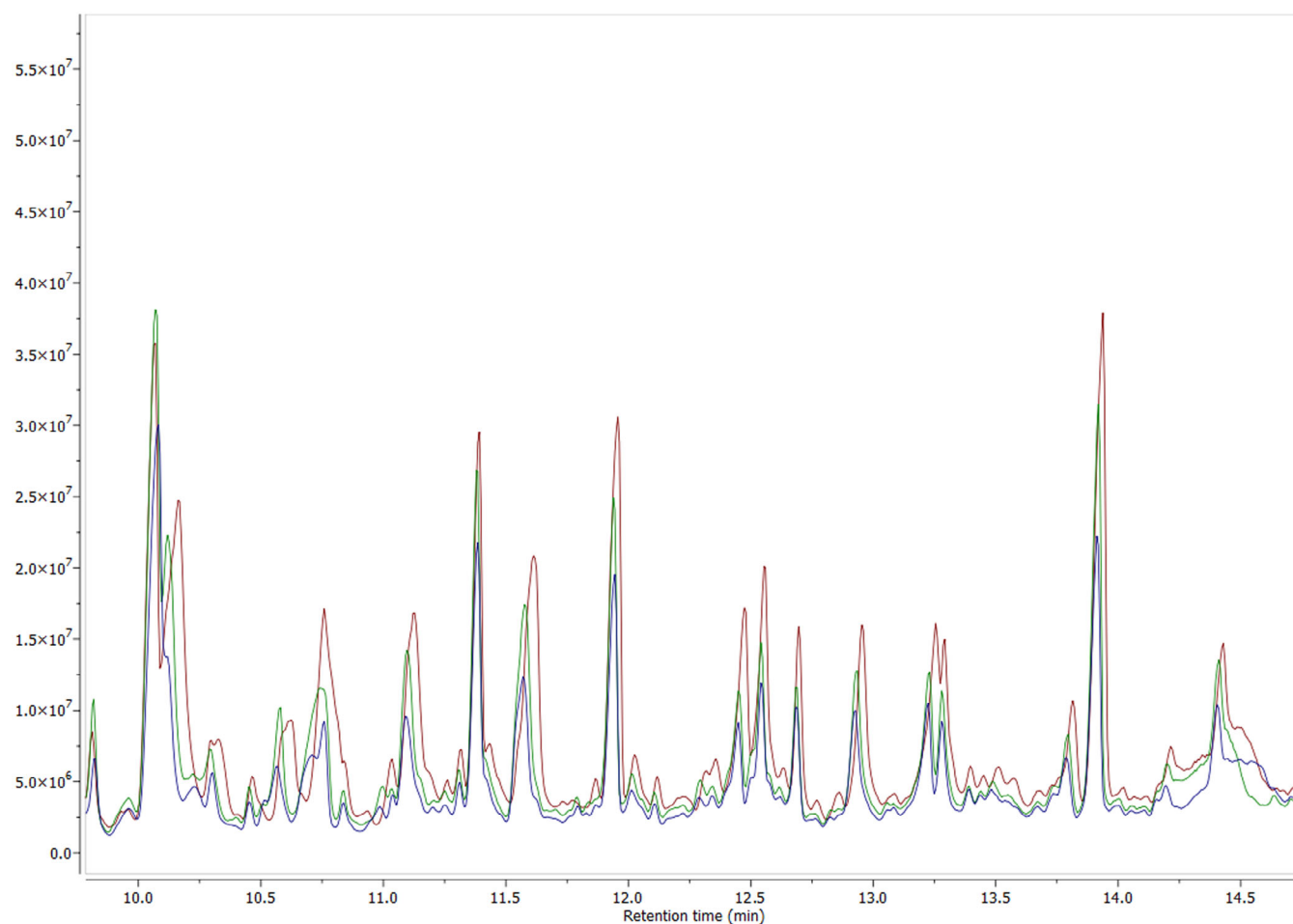


FIGURE 2 Comparison of chromatograms between samples O7 (green), O8 (blue), and O9 (red)

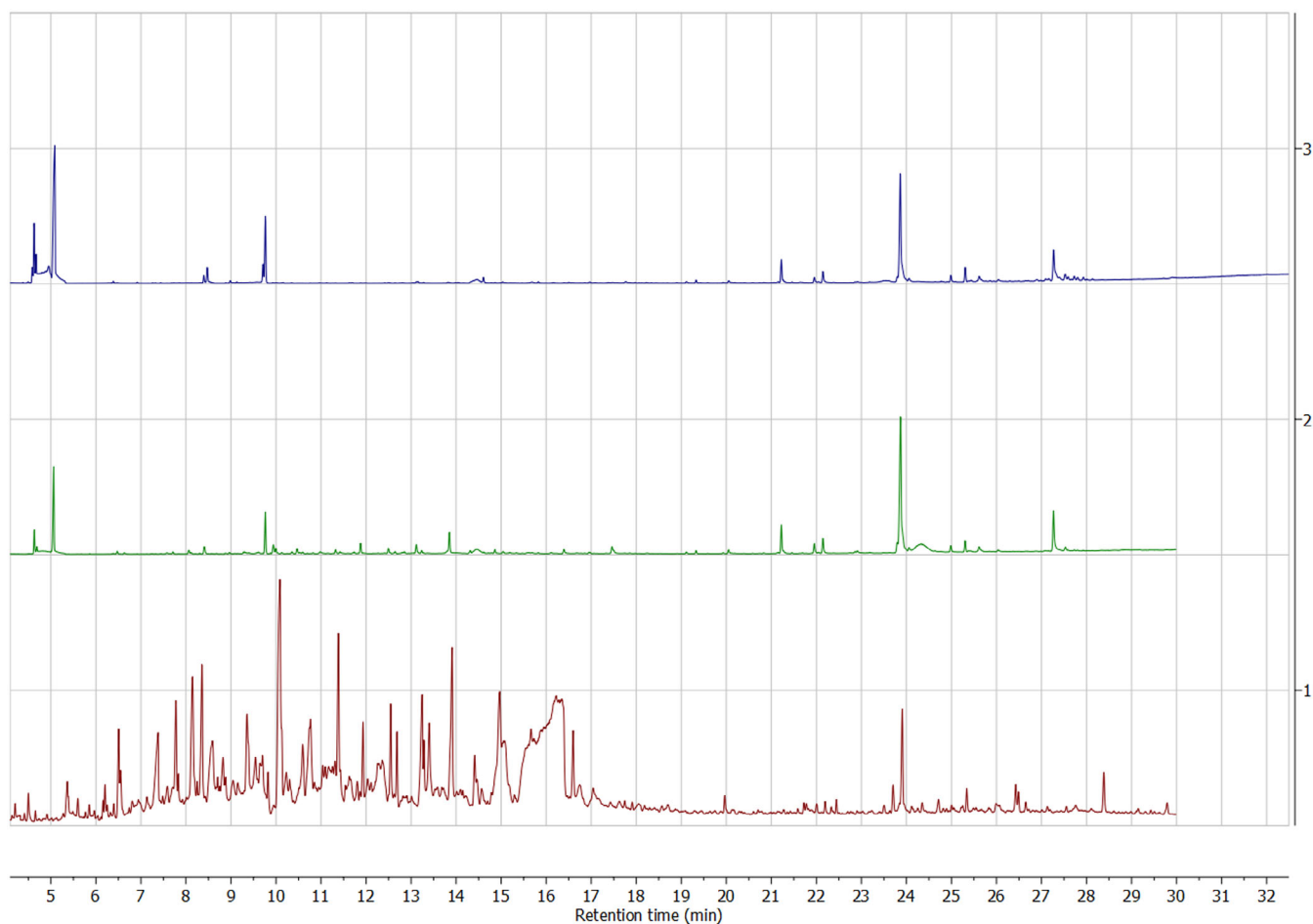


FIGURE 3 Comparison of chromatograms from selected spruce samples (top to bottom: PI1, PI4, PI7) across oxygen levels (21%—upper chromatograph, 15%—middle chromatograph, and 10%—lower chromatograph)

with the extraction and analysis of chemical compounds to obtain accurate and repeatable data, was also evaluated. Several types of wood-based materials were burned. The effluents were captured on glass wool (GW) filters and PUF plugs, extracted into acetonitrile and *n*-hexane and analyzed using gas chromatography coupled with a mass spectrometer (GC-MS). The methods to extract and analyze chemical compounds from fire effluents were based on a variety of work done by several authors.^{20–24} The method of analysis focused on semi-volatile species (lowest boiling point $\sim 60^{\circ}\text{C}$), while common toxicant gases (CO_2 , SO_2 , CO , etc.) were not monitored.

3 | EXPERIMENTAL DATA

3.1 | Construction of MCACC

The chamber follows ISO 5660-5 standards (37 by 33 by 30 cm).

The stack used was shorter than the standard, measuring at 500 mm as opposed to the prescribed 600 mm. However, no afterburning was observed during the experiments. The gases were fed into the enclosure at total 2,4 L/s. The concentration of the

oxygen in the supplied air/nitrogen mix is controlled by balancing the input flows of compressed air and pure nitrogen. The oxygen concentration is additionally measured using a sampling point close to the test specimen (roughly 50 mm distance) and at the top of the stack. The sampled gas is first led through a GW filter to eliminate particles larger than $1\ \mu\text{m}$. After that, it is led through a drying column filled with silica gel and then to CM 42991 oxygen concentration measuring sensors. Effluent sampling point was positioned at the top of the stack. The effluents were sampled at 3% of total flow, that is, 0,072 L/s and led through a GW filter and a PUF plug.

3.2 | Burning experiment

The cone heater was calibrated using a heat flux meter in accordance with ISO 5660-1.

The sensors for measuring the oxygen concentration were calibrated against a paramagnetic reference to establish their sensitivity and drift. Because the measurement of the oxygen concentration with these sensors is based on the oxygen partial pressure, it is expected that the reading depends on absolute pressure of

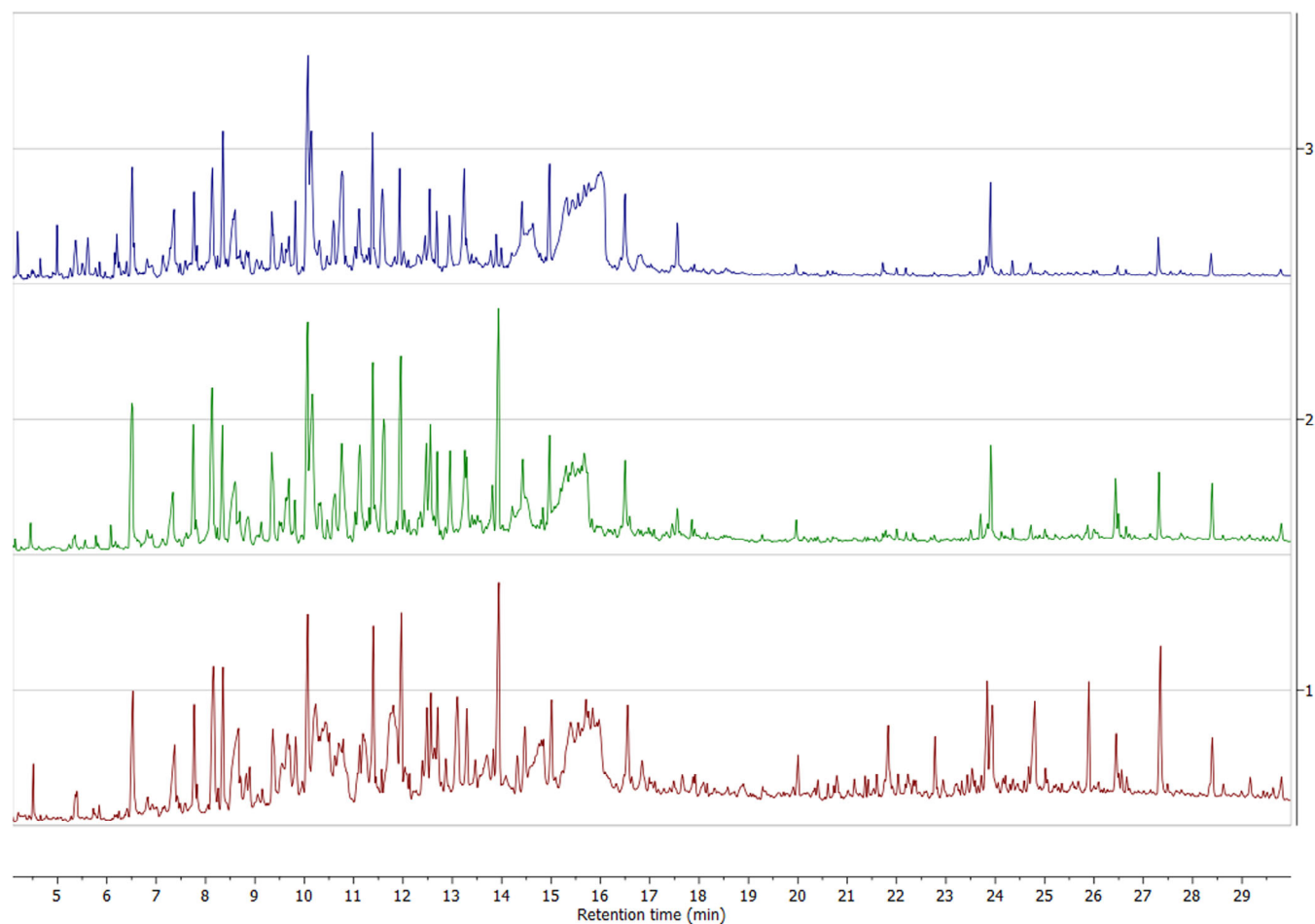


FIGURE 4 Comparison of chromatograms from acetonitrile extracts of samples PI7 (3), O7 (2), and PB7 (1) obtained at 10% oxygen level

the gas in the chamber with the oxygen sensor. Before the experiment, the oxygen concentration was measured in the surrounding clear air. The assigned value was 20.95%. Readings of the sensors were corrected to the initial value each time at the beginning of the experiment. The correction was 1% or less, and the overall accuracy of the oxygen concentration measurement was assessed as $\pm 0.5\%$ or better.

Other parameters measured as part of the ISO 5660-5 test (gases via FTIR, HRR etc.) were not monitored as part of our experiments.

Several common wood-based materials were considered for initial testing, with spruce, particle board, and oriented strand board (OSB) chosen in the end. Samples were cut from a single board into standard 100 mm by 100 mm by 20 mm pieces (additional sample data in Data S1). The chamber was closed, and the oxygen level was allowed to equilibrate to the chosen concentration with the heater shuttered. The chamber door was then briefly opened and the sample mounted. The chamber was allowed to equilibrate again, at which point the shutter was opened, and the samples were exposed to 50 kW/m² radiation from the cone heater for 20 min. The desired oxygen concentration at the level of the sample's burning surface (21%, 15%, or 10%) was achieved by mixing compressed air (local ambient air compressor) and nitrogen (technical grade, Messer) in the correct ratio. Fire effluents were captured using a GW filter for particulates and

precleaned ORBO 1000 PUF plugs (Supelco) for semi-volatile substances. Sampling was carried out through a sampling port on the stack (see Figure 1) by pumping the effluents through the filter and the plug at 0.072 L/s (3% of the in-flow) for the full experiment duration (20 min). After collection, the plugs and filters were kept refrigerated at nominally -20°C until further processing. Three samples of each material were burned at every oxygen concentration (Table 1).

3.3 | Extraction of smoke effluents for GC-MS analysis

The GW filter and PUF plug with collected smoke effluents were cut in half, with each half further snipped into smaller pieces. One of the pair was added to 15 mL of acetonitrile (Sigma-Aldrich Chromasolv, LC-MS grade), while the other half was added to 15 mL of *n*-hexane (Sigma-Aldrich Chromasolv, 95%). Solvents were chosen to maximize the variety of extracted compounds. The samples were put into an ultrasonic bath for 15 min at room temperature, decanted, replaced with fresh 15 mL of solvent, and sonicated for a further 15 min. Both fractions of the extract were combined and concentrated on a rotary evaporator to 10 mL, then further concentrated under a gentle flow

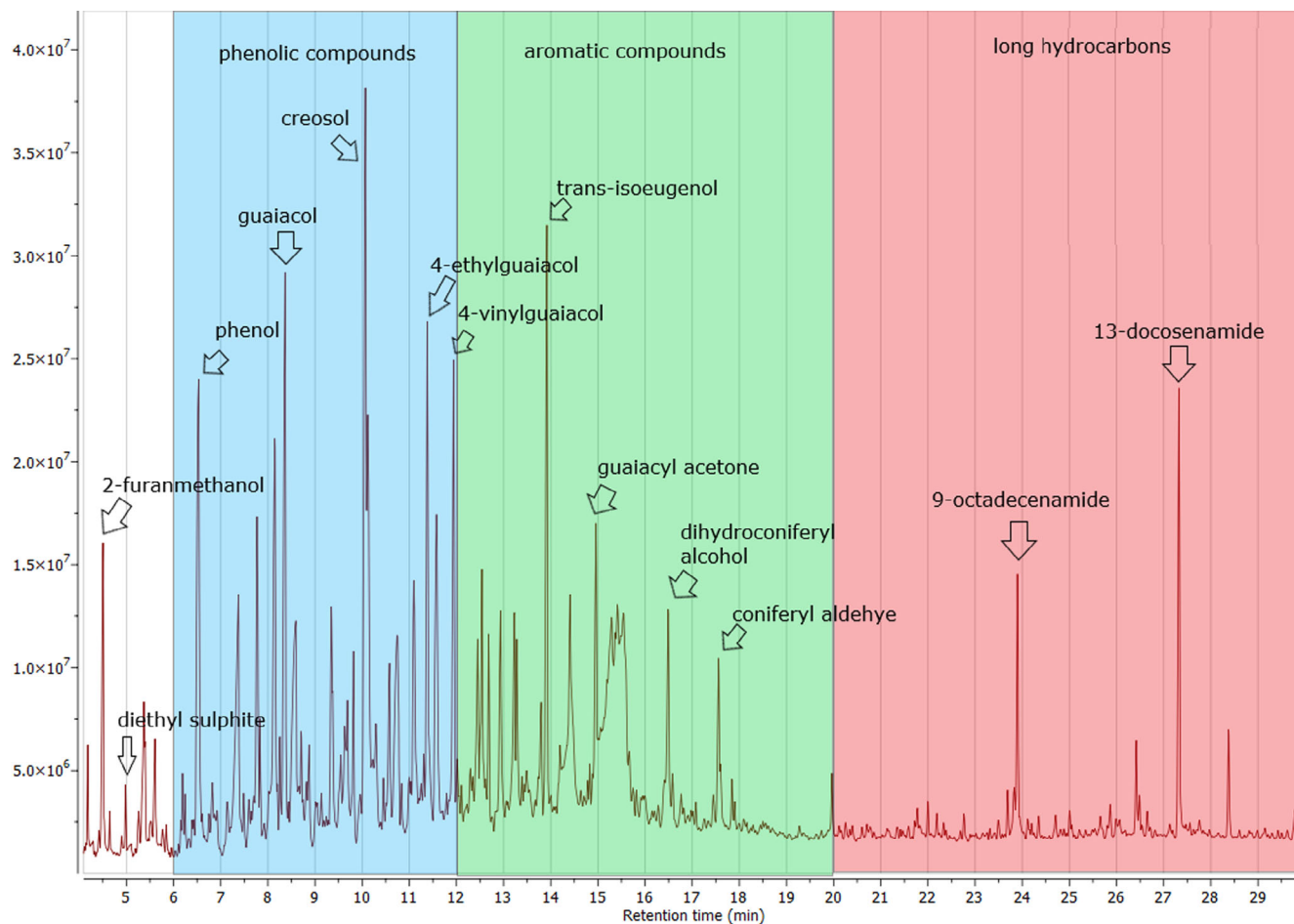


FIGURE 5 Chromatogram of acetonitrile extract of O8 with some reference peaks indicated

of nitrogen to 1 mL final volume, transferred to a crimped vial, and frozen until the analysis.

3.4 | Instrumentation and GC-MS conditions

An Agilent 5977B single quadrupole GC/MSD was used for analysis of the extracts. The GC separation was conducted using an Agilent J&W HP-5 ms Ultra Inert column. Analysis conditions were as follows: carrier gas He (1.2 mL/min); injector temperature 300°C, and transfer line temperature 300°C. 1 μ L splitless injection was carried out by a Gerstel autosampler. The GC temperature conditions were as follows: initial temperature 50°C (hold: 90 s), heated to 325°C (ramp: 10°C/min, hold: 60 s).

Compound determination was undertaken using Agilent Masshunter software, utilizing the 2018 NIST mass spectrum database. Compounds were manually verified when needed using the NIST 2018 and massbank.eu spectral databases.

4 | RESULTS AND DISCUSSION

The addition of a sampling port to the stack allows for simple monitoring of fire effluents produced. The position at the top of the stack

allows for sampling of effluents undiluted and unreacted with the ambient air, while keeping the reaction path long enough for the gases to react and cool somewhat.

Exposing samples to cone radiation at different oxygen levels: 21%, 15%, and 10% produces considerably different effluent profiles, fairly reproducible across samples as determined from PUF capture plugs with GC-MS. The major differences between the profiles were mainly due to the oxygen levels and consequently the mode of combustion (flaming or smouldering, for complete sample breakdown see Data S1) and less due to the nature of the material (Figures 3 and 4). As expected, all samples combusted into flame at 21% oxygen and all smouldered at 10%. Most samples also flamed at 15% oxygen, with PB5 being the sole exception. As Figure 4 shows, the mode of combustion has the greatest impact on the chromatogram, as most monitored compounds are not formed in appreciable concentrations until there is a prolonged period of smouldering. However, it should not be overlooked that even samples that did not smoulder at 15% oxygen produced a consistent and marked increase of measured products (mainly unsaturated hydrocarbons) when compared to flaming samples exposed at 21%. The integral counts of major peaks among closely related samples were generally within 100% of the mean on smaller peaks to less than 30% of the mean on higher ones, with very similar peak shapes and positions (Figure 2). The difference between

different materials being rather insignificant and quite possibly within sample variance (Figure 4) is perhaps regrettable if tentatively expected, but it shows considerable repeatability of the obtained products. The combustion products identified from the chromatographs were compounds expected from the pyrolysis of wood and internally consistent.²⁵

At atmospheric (21%) oxygen levels barely any volatiles are noticed on the chromatograph (Figure 3). Some major peaks are noted on Figure 5, which serves as a guiding reference. Among the few major peaks are diethyl sulphite (retention time [RT] cca. 5 min), which is likely the final form of most sulfurous compounds present in the wood; diethyl succinate (RT cca. 10 min), which is likely created by what is technically dry distillation of resinous wood; trans-iso Eugenol (RT cca. 14 min), which is a common component of any wood smoke;²⁵ and various derivatives (mostly amides) of long hydrocarbons (9-octadecenamamide, 13-docosenamide, 1-octadecanol, palmitoleamide, hexadecanamamide etc.; RT above 20 min). Other compounds like phenols (RT between 6 and 12 min) are also able to be determined, but their concentrations are low. Short of diethyl succinate and trans-iso Eugenol, quantities of most of the other compounds are very similar across samples and even oxygen levels, which is expected, as the mechanism of their creation is independent of the concentration of oxygen present.

Running the experiment at 15% oxygen levels, we noticed increased levels of unsaturated hydrocarbons in the captured effluents (Figure 3). Along the previously mentioned compounds, we identified noticeable amounts of aromatic compounds, mainly phenol and various phenolic derivatives, among them 2-methyl phenol, 3-methyl phenol, 2-methoxy phenol, 2-ethyl-phenol, 2,4-dimethyl phenol, 2,5-dimethyl phenol, catechol, eugenol, vanillin etc. (RT between 6 and 12 min). Several polyaromatic compounds such as naphthalene, methylnaphthalene, 9-fluorenone, anthracene, phenanthrene, etc. were also determined as present in low amounts. Some samples smouldered for quite a while or did not flame at all, and the concentrations of determined compounds were uniformly higher in their chromatograms.

At oxygen concentration of 10%, the concentrations of aromatic and polyaromatic compounds (RT between 6 and 20 min) increase dramatically (Figures 3–5). Roughly speaking, we see an increase in integral counts of approximately ten times every time we go down an oxygen step. Full compound lists and chromatograms can be found in Data S1. However, as some concentration levels are rather uniform across samples, further experimentation needs to be done to determine whether an equilibrium or saturation is reached during the exposure of the PUF plug. There might also be concerns about surface-catalyzed reactions on the heated PUF plug. However, the abundance of unsaturated compounds detected and the inertness of polyurethane foam make that unlikely.

5 | CONCLUSIONS

In conclusion, the modified cone calorimeter allows reproducible creation of fire effluents under controlled burning circumstances. The biggest variance is found in samples exposed at 15% oxygen, which is expected, as this oxygen concentration is close to the limiting oxygen

concentration for a variety of solid materials, including wood.²⁶ Samples which undergo smouldering for a longer time contain more semi-volatile effluents than samples that catch fire faster. Some of the difference might also be attributed to incomplete burning, as the borderline condition might lead to lower flame temperature and other differences in unmonitored parameters, and as such, chemical reactions taking place. Selected methods of capture and analysis were also proven satisfactory, as they proved useful in gathering and identification of a variety of compounds present, including a multitude of known irritants, carcinogens, and otherwise harmful compounds. The toxic potential of these compounds needs to be evaluated with a semi-quantitative or quantitative approach, but repeatable detection is the first step on this way. Some minor questions and fine-tuning are still open, but the modified ISO 5660-5 cone calorimeter shows considerable promise as a potential tool in determining nonacute toxicity of smoke from various materials on the benchtop and ultimately contribute data for the numerical modelling of toxicity of fire effluents in real buildings.

CONFLICTS OF INTEREST

The authors declare they have no competing interests.

DATA AVAILABILITY STATEMENT

The authors confirm that the data supporting the findings of this study are available within the article and its supplementary materials.

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SUPPORTING INFORMATION

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